National Bureau of Standards

TECHNICAL NEWS BULLETIN

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# Detection of Isotopes by Tracer Micrography

A new method for the more effective tracing of radioactive isotopes in materials in which they have been intentionally introduced has been developed by L. Marton of the National Bureau of Standards with the cooperation of P. H. Abelson of the Department of Terrestrial Magnetism, Carnegie Institution of Washington. In this procedure, by means of a magnetic focusing arrangement, the radiation given off by a radio-isotope within a sample material is made to form an image of the emitting surface upon a photographic plate. This image may then be used in studying the distribution and concentration of the radioactive element present in the sample.

In many chemical, biological, biochemical, and other fields of research, there is a growing application of the method of tracers, in which the isotope of a given element is used as an indicator to tag or label certain groups of atoms so that they may be distinguished from other atoms of the same kind. Identification of tracer elements is at present greatly facilitated through the use of radioactive isotopes, which, because of recent developments in atomic energy, are now available in large quantities and are relatively easy to detect through their radiations.

In the well-known method of radio autography a radio-isotope is introduced in a biological or other system, and the distribution of that particular element within the system is determined by bringing the sample in close contact with a photographic emulsion. This method lacks resolving power because, even in case of perfect contact of the sample with the emulsion, the circle of confusion from every point of emission is so great that details less than a tenth of a millimeter are very difficult or impossible to distinguish.

In order to improve the resolution of this tracer method, it was decided to use electron optical image formation for determination of the distribution of a radioactive element within a given sample. This process, which may be called "tracer micrography", is based on the emission of high-speed electrons (beta rays) by many tracer elements and the use of magnetic lens elements to form an image on a suitable recording surface.

In the absence of any means for correction of the chromatic aberration of electron optical lenses, the first micrographs were limited to those elements that emit electrons of uniform speed. After some attempts with columbium,[95], yttrium [87], strontium [85], strontium [87], protactinium [233], and gallium [67], the latter was selected for the initial tests. Gallium chloride, prepared by chemical separation from zinc, was bombarded by heavy hydrogen nuclei in the cyclotron at the Carnegie Institution, and the solution was evaporated drop after drop on a ¼-inch tantalum disk. Radiation emitted from the surface of the disk, upon passing through a magnetic lens consisting of a small ironclad coil with Armco iron pole pieces, was brought to a focus upon a photographic film at a distance of about 3½ inches. An image of the tantalum disk was thus obtained showing radioactive areas. The conditions were selected

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so that a linear magnification of 2 was obtained.

For calibration of the instrument, the photographic film was replaced by a Geiger counter, and the lens current necessary to produce a maximum number of counts in unit time was determined for radiations of varying velocities. This establishes the focusing current for a given type of radiation.

In preliminary experiments with samples of different concentration and thickness of the radioactive layer, exposure times ranged from 2 to 12 hours according to the age and concentration of the sample and the numerical aperture of the lens. It was found that micrographs with good definition were obtained consistently when the layer was sufficiently thin to avoid considerable self-absorption. The best resolving power attained so far has been about 30 microns.

The simplicity of this method, both in apparatus and technic, is one of its more important features. Vacuum requirements are very moderate, as the mean free path of the electrons is large in comparison with the apparatus dimensions, even at forepump pressure.

Further improvements in tracer micrography are expected through after-acceleration of the beta particles by means of an homogeneous electrostatic field. Such after-acceleration may well result in reduced exposure time and in better resolution due to a reduction in spherical aberration. A further reason for after-acceleration is that chromatic aberration, which is always present, even in sources emitting particles of uniform speed, can be markedly decreased if the accelerating potential is at least comparable in magnitude to the energy of the primary emission.


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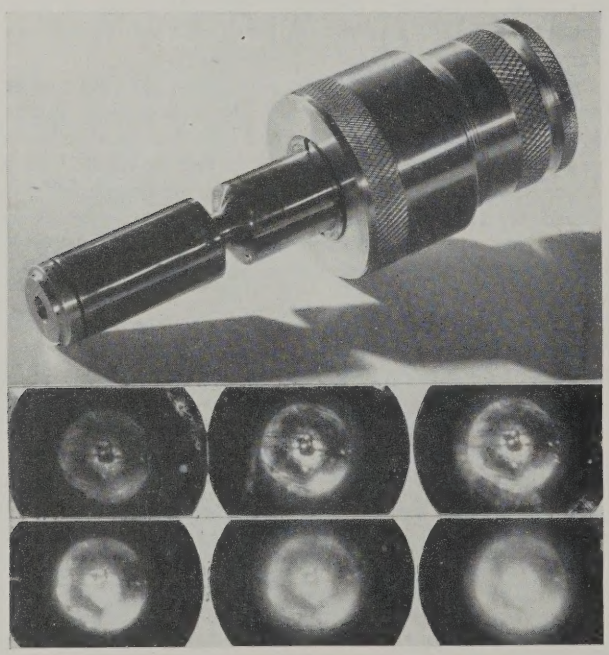
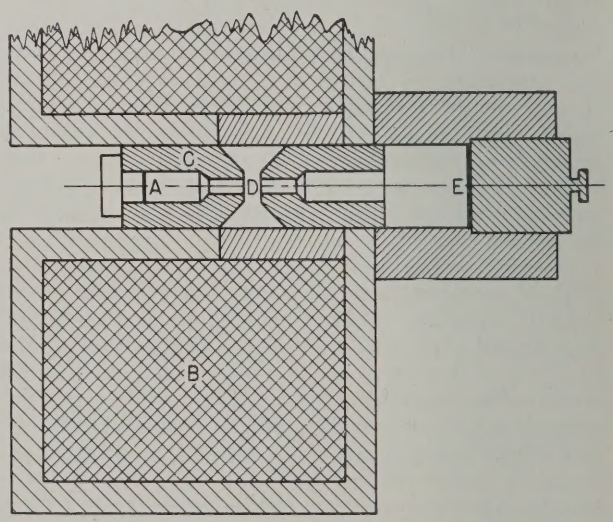





**Location of radioactive tracer elements is facilitated by a new electron optical method, "tracer micrography." Electrons emitted by a radioactive sample at *A* (right) are focused through the magnetic field *D* of a magnetic lens onto a photographic film at *E*. The pole pieces *C* and film holder are shown (upper left) removed from the magetic coil *A*. Photographic images (lower left) reveal the distribution of radioactive areas. Improved resolution in the lower row was obtained by reduction in size of aperture.**

# The Mass Spectrometer in Gas Analysis

The mass spectrometer, one of the newer analytical instruments, has shown itself to be a remarkably useful apparatus in the analysis of gases and complex mixtures of gases. Many industries, gas, petroleum, chemicals, and rubber among others, rely heavily on gas analysis, and any improvement in methods or apparatus is of far-reaching importance. Although established chemical methods have long yielded valuable information concerning compositions of gases, they have serious limitations. The mass spectrometer offers an entirely new and different instrument for this type of analysis. It can, in one operation, resolve a very complex mixture of gases in a few moments of instrument time, although subsequent computing may require hours or even days, depending upon the complexity of the mixture and the method of computation employed.

The mass spectrometer offers the nearest approach to a real qualitative and quantitative system of gas analysis that has yet been devised. Gas analysis is in some respects the most curious branch of analytical chemistry. No other kind of analysis of equal importance is so totally devoid of a definite system. When a mineral is analyzed, there is a qualitative system for discovering the elements and radicals present, and a quantitative system for determining the amounts of each. But when a gas mixture is analyzed by the conventional volumetric chemical methods, the kinds of gas present are deduced from typical reactions, which themselves are designed to disclose the *amounts* of these gases. In many cases the identifying reaction is not sufficiently specific to resolve the mixture to its actual components. Thus combustion methods are incapable of identifying or estimating more than two hydrocarbons of a related series in any one sample.

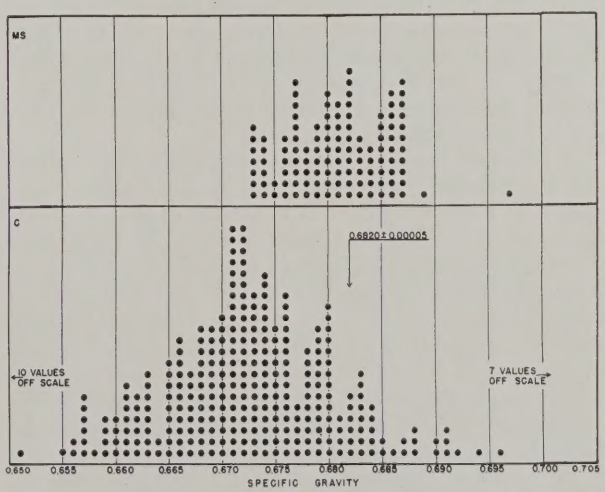
The mass spectrometer separates various gases on the basis of the actual weight of the ions characteristically produced on dissociating each molecular species by electron bombardment. In many instances the separation affords a clear-cut identification of each component of the mixture. Usually the quantitative resolution may be achieved by proper mathematical treatment of the spectrometer data; and often the qualitative and quantitative processes are simultaneously conducted as in the older chemical methods.

Both the mass spectrometer and the conventional volumetric chemical methods have recently been used by a group of cooperating laboratories in the analysis of a standard sample of natural gas. Twenty laboratories possessing a total of 21 spectrometers participated in the spectrometric tests, and 30 laboratories in the chemical tests. This represents one phase of a study of existing methods of gas analysis by means of a Nation-wide cooperative analysis of standard samples, which the Bureau is making in cooperation with a subcommittee of the American Society for Testing Materials. (These samples are not to be confused with the regular standard samples issued by the Bureau for purchase, but are carefully prepared gas samples furnished to cooperating laboratories). Reports of this cooperative analysis [1] give for the first time a picture of the actual state of gas analysis in this country with respect to two of the three most important methods of analysis, the third method being fractional distillation.

The analytical data have been translated from extensive tabular form to a set of frequency-distribution tests that reveal at a glance the entire story both in general and in essential detail. A typical frequency distribution test illustrates to some extent the trend of the whole study. The values given in the upper section marked MS were calculated from analyses by the mass spectrometer, and those in the lower section C from chemical analyses. In this plot, every circle represents the specific gravity calculated from one analysis of the standard sample. The abscissae are values derived from the analyses, and the ordinates indicate the frequency with which they occur. For example, reading the upper (MS) section, it is seen that 7 analyses yielded the value 0.673, 6 gave 0.674, 2 gave 0.675, and so on. The known measured value was $0.6820 \pm 0.00005$.

This plot by no means gives the complete comparison derived from this study. In general the results are scattered considerably more than had been expected. The lack of reproducibility between laboratories clearly demonstrates for the first time the real need for standardization of analytical methods and procedure in the field of gas analysis. Although the spectrometric values show better reproductivity and closer agreement with the known composition than do the chemical values, further improvement is believed possible. As the method is new and has not yet been subject to great variations of apparatus and procedure, efforts are being made to establish a standard procedure that will lead to even better results with the mass spectrometer.

[1] Martin Shepherd, NBS J. Research **38**, 19 (1947) RP1759; **38**, 491 (1947) RP1789.




**Frequency distribution tests of data from cooperative analyses of a standard gas sample by two methods reveal a need for standardized methods and apparatus.**

**Entire heating systems were installed in this test bungalow, constructed by the Bureau for studies of heat transfer phenomena, to determine performance of various heating devices designed for low-cost housing. The bungalow is now enclosed with an insulated shell so that constant temperatures can be maintained outside of the house.**

# Temperature Distribution in a Test Bungalow

Uniformity of temperature throughout houses is a tacitly accepted American ideal of heating. It is probably never attained in practice, and departures depend upon the design and construction of the house and upon the characteristics of the heating system or device used. Distribution of warmth in houses has usually been judged only qualitatively by individual engineers on the basis of personal experience or observation. Quantitative data have been limited almost entirely to laboratory tests on parts of the system. In order to obtain data on the temperature distribution in an entire house, a series of heat distribution tests of several types of heating systems were conducted at the Bureau by Richard S. Dill and Paul R. Achenbach.[2]

A full-scale house in which complete heating systems can be installed was constructed at the Bureau. Designed as a test bungalow, it is similar in plan to house B described in Technical Bulletin No. 4, "Principles of Planning Small Houses," issued by the Federal Housing Administration. It has four rooms and bath, with a central hallway. The walls are conventional in construction, consisting of ½-inch gypsum board on the inside, and 2- by 4-inch studding with sheathing and lap siding on the outside, separated by a layer of building paper. All the windows are double hung, with the exception of that in the bathroom, and one of the two in the kitchen. The double floor of 1-inch pine includes building paper between the subfloor and finish floor. For the heat distribution tests the walls were not insulated, but a 2-inch blanket of wood-fiber insulating material was laid over the ceiling. Weather-stripping was not provided for windows or doors.

During the tests, data on temperature conditions inside the bungalow were recorded by various types of instruments. Heat-transfer coefficients for floors, side walls, and ceilings were measured by the Nicholls type of heat-flow meters fastened to the surfaces. Thermocouples located at various levels of the rooms, on top and bottom of the floor, on the inside and outside of the walls, and in the basement and attic air, gave a complete record of temperature conditions. Observations were made while heat was being supplied in turn by an experimental electric heater, an oil-burning warm-air furnace, a jacketed gas-firing space heater, a jacketed oil-fired space heater, a single gas-burning gravity floor furnace, two gas-burning gravity floor furnaces, a gas-burning floor furnace with forced circulation, an oil-burning gravity floor furnace, and a conventional gravity hot-water heating system.

From the standpoint of comfort, the temperatures from the floor to 5 feet above the floor are more significant than the temperatures at higher levels. The tests showed that the average temperature differences produced in the test bungalow between the 2-inch level and the 60-inch level, with continuous forced circulation of air through a plenum chamber, with a forced-circulation gas floor furnace, and with a gravity hot-water heating system, were less than 10° F, when the outside temperature was 32° F. The temperature differences produced in this same zone by all the other

devices tested ranged from 14° to 18° F at comparable outside temperatures.

The average horizontal temperature differences between rooms at all levels of measurement ranged from 2° to 4° F for the several floor furnaces and for the gravity hot-water system. For the electric warm-air furnace and the oil-burning furnace, when attached to a plenum chamber, these differences were nearly as small for the 2-, 30-, and 60-inch levels, but were considerably greater in the upper levels of the house. The temperature difference between rooms was greater for the space heaters in the living room than for the other heaters tested. This effect can probably be attributed to the fact that the rooms other than the one containing the heater are warmed by the overflow of heat from the living room through the doorways. Except for the space heaters, the average horizontal temperature differences from 2 inches to 60 inches above the floor did not increase with lower outside temperatures.

Certain types of heaters may be suitable for houses of one construction and not suitable for other constructions. For example, a high temperature near the ceiling would not cause much additional heat loss if the ceiling were well insulated, or an insulated floor might permit the use of heaters that do not deliver warmed air at or near the floor level. Furthermore, some types of heaters that provide comfort in areas where extremely low temperatures do not occur may not provide comfort in colder climates.

Although standards of performance in the field of vertical or horizontal temperature differences in residences have not been established, the Bureau tests indicated that the air temperatures in the living zone of all rooms should be in the range from 65° to 80° F for comfort when heated by the conventional types of heating systems. A floor temperature of 60° F, when continued for 1 hour or more, was found to be too low for foot comfort; when a floor temperature of 65° F was continued for the same period, discomfort was no longer apparent. It was further observed that air temperatures of approximately 85° F around the level of the head became oppressive, especially if the air had an appreciable velocity.

The results obtained with the devices used in the test bungalow show need for further improvement in the design of house construction and heating systems in order to provide comfort in all rooms of basementless houses. A more uniform temperature can probably be obtained either by the use of more insulation in the house elements, by improving the heat-distribution systems, or by a combination of both.

[2] The complete technical report on this project is contained in the National Bureau of Standards Building Materials and Structures Report BMS108, Temperature distribution in a test bungalow with various heating devices.

# Protection Against X-Radiation

Scientists at the National Bureau of Standards are engaged in an extensive program for determining the effectiveness of concrete as a protective barrier against million-volt wide-beam X-rays, whose use is increasing in medicine and industry. At the present time, exact wall thicknesses and most desirable types of construction necessary for maximum short-wavelength X-ray protection are not definitely known. One of the basic aims of the new project is to collect data from which the highest degree of protection with the lowest possible cost of installation can be calculated.

Industrial uses for million-volt X-ray equipment were greatly increased during World War II when industrial engineers turned to it as a device for detecting flaws in all types of metal. In particular, broad-beam X-rays, which allow simultaneous examination of wide areas, came into extensive use. Although broad-beam X-rays, ranging in diameter up to 4 or 5 feet, are not essentially different (except for the amount of surface covered) from narrower beam radiation, they pose a special protection problem.

When a broad X-ray beam enters a thick concrete wall, it is scattered and rescattered many times with the result that a considerable fraction of the beam emerges on the other side, endangering personnel. Quantitative information on the amount of this scattering is not yet available.

Institutions using million-volt equipment have been seriously concerned with this phenomenon, and in order to assure a wide margin of safety have constructed walls of exceptional thickness. Field attempts to make measurements establishing optimum thicknesses and construction characteristics involve so many variables that the entire project, except for final testing, must be carried out in the laboratory, where the variables affecting radiation hazards can be simulated, controlled, and analyzed.

Industrial X-rays are used in three ways for metal examination. Radiographs are obtained by directing beams of X-rays through a metal object and recording the shadow lines on photographic plates. Many examinations of small parts are made by a fluoroscopic screen. A relatively new technic, X-ray diffraction, is a method for studying crystalline structure, where the X-rays are diffracted by the crystals within the metal and are recorded photographically or by means of a Geiger counter.

To determine protection standards, a 1½-million volt X-ray machine in the Bureau's laboratories has been converted so that it will produce beams of varying widths. It is mounted so that it can be directed downward into a "radiation pit" about 6 feet square and 20 feet below the target of the X-ray tube. Special instruments, designed to explore the strength of the radiation in the pit, can be shifted to various positions by remote control. Both the control and the X-ray reading equipment are operated from a central control room surrounded by 18 inches of concrete and located 75 feet from the radiation pit.

During the investigations, test slabs of concrete each

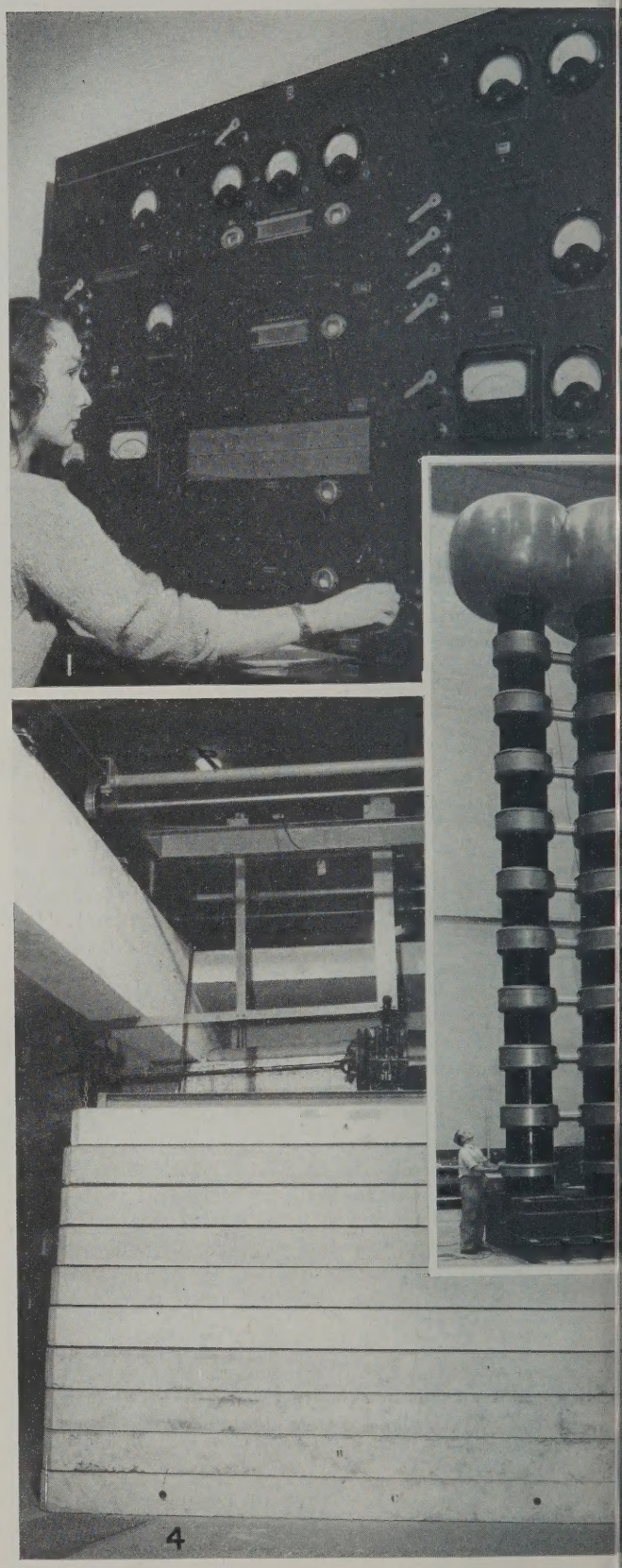
weighing 2½ tons and measuring 6 inches in thickness are used. These slabs, when placed over the mouth of the pit, one at a time, enable investigators to vary the protective barrier from 6 inches to 5 feet in thickness. As space and weight are of critical importance in many installations, arrangements have also been made for producing "sandwiches" of lead and concrete in alternate layers. At present, the best proportions between these two materials are entirely unknown.

Studies of radiation hazards have been carried on continuously at the Bureau since 1913. At that time the early type of gas-filled X-ray tube was used. Though voltages were low, long exposures may have been responsible for the many cases of serious burns on record for that period. About the time of the first war, the new high-vacuum Coolidge X-ray tube came into use, operating at higher voltages, with radiations of correspondingly greater powers of penetration. Reduced exposure times eliminated some of the hazard to the operator. But accompanying this was the increased penetration of the X-ray due to higher voltages and the greatly extended use of X-rays with the increased versatility of the equipment. X-ray tubes for diagnostic processes were built for operation around 90,000 volts. Soon after this came the introduction of the superficial therapy X-ray tube operating at potentials of 140,000 volts.

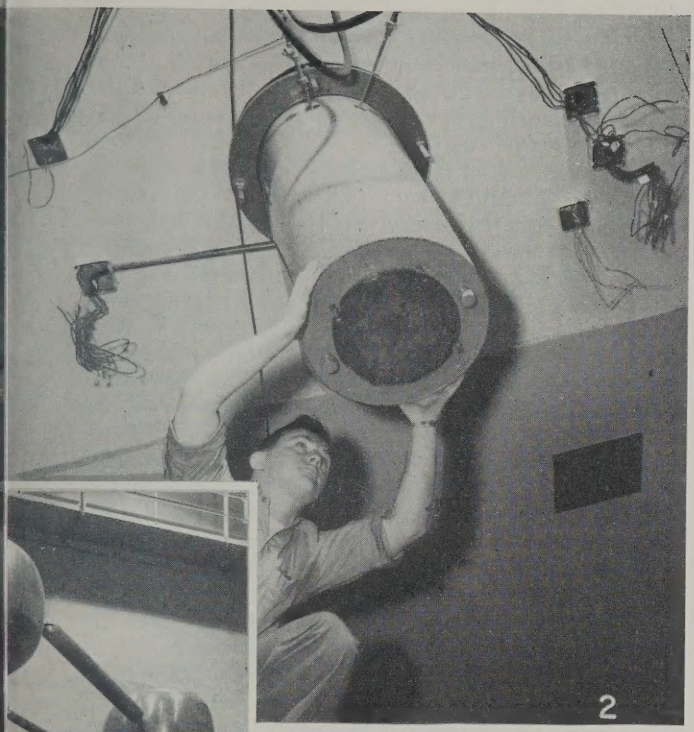
In the early twenties there appeared the first tubes for deep therapy use operating at voltages of 200,000 and 250,000. Here the hazard was greatly increased, as was the problem of protection. In 1927, urged and supported by all of the radiological societies and the American Medical Association, the Bureau undertook a broad expansion of its activities in the X-ray field. These were centered about two major problems: (1) That of measuring X-ray dosage and (2) the study of X-ray protection hazards and formulation of principles to be used in their elimination.

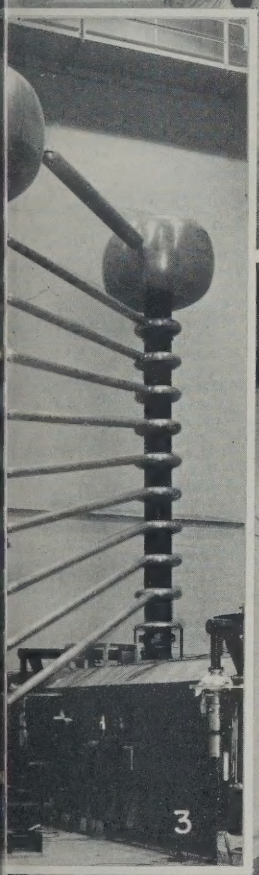
For equipment operating up to 200,000 volts, lead then afforded the best means of protection. A room used for diagnostic purposes needs to be lined with 1/16 inch of lead to prevent escape of the rays to surrounding rooms with consequent injury to the occupants. Correspondingly, in the deep therapy installations the walls of the room must be surrounded with 3/16 to ¼ inch of lead. Because ¼-inch lead weighs 16 pounds per square foot, the installation of such protective shields becomes a massive and costly project. In the early days, such protection was almost mandatory, as the X-ray tubes were operated in the open with penetrating X-rays emitted in every direction.

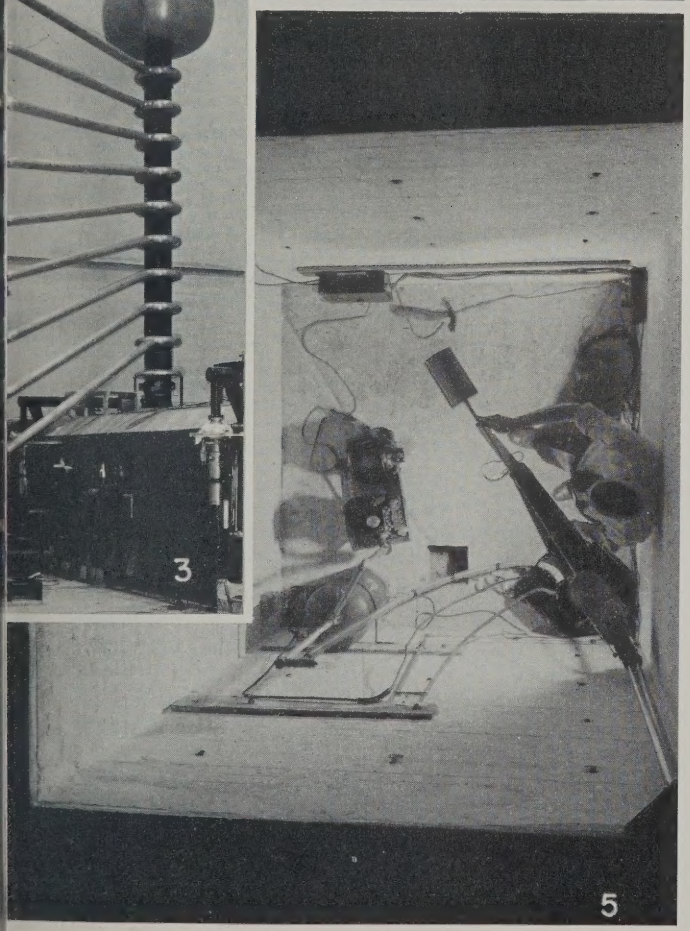
One of the Bureau's first problems was to study ways




**X-ray protection studies have been conducted at the National Bureau of Standards continuously since 1913. Present X-ray equipment at the Bureau (3) includes a generator (large central column) and X-ray tubes (right) capable of producing 1½-million-volt X-rays. This apparatus is operated through a central control station (1) that is equipped with an interlock system to insure proper sequence of operation. The target end of the X-ray tube (2) extends into the target room, protected by massive walls of concrete.**







and means for improving protection in the immediate vicinity of the X-ray tubes in such a way as to reduce the protection necessary in the walls. As a result of these early studies, it was realized that a relatively small weight of lead could be placed closely around the X-ray tube allowing the X-rays to escape only in the direction in which they would be utilized on the patient. By doing this, lead protection in the remaining walls of the room could be reduced 50 to 75 percent except for the small portion of the walls or floors in the path of the main beam. This meant immense savings in the initial cost of X-ray installations.

At about the same time, studies were undertaken to determine the efficiency of such auxiliary protective devices as lead aprons, lead rubber gloves, lead glass windows, and wall plasters made of material loaded with lead or barium. Some of these devices were found to be effective, while others were not. For example, lead glass goggles for the protection of the eyes were found to offer little or no protection. In fact, they proved to be more of a hazard than a protection, because they gave the user a false sense of security.

Lead glass has always been a necessary adjunct to an X-ray protective installation. This is particularly true in therapy work where the operator of the machine must remain outside the treatment room and yet have a good view of the room through a window or some other device. Such windows were made of glass loaded with lead. Many of the glasses sold for protective purposes were found to contain so little lead as to be of negligible value. Others have adequate quantities of lead but were so discolored by the addition of lead that they are unsuitable for window purposes. Up to about 1930 practically all of these glasses were imported from Europe. Under the impetus of a broad study of X-ray protective glasses by the National Bureau of Standards, American industry was encouraged to undertake the development and production of such materials. This progress was piloted from the X-ray point of view by the Bureau and, as a result, this country is now able to produce the finest grades of lead protective glasses available anywhere in the world.

Beginning about 1930, X-ray tubes for operation up to 400,000 volts and higher were developed and began to come into use for both medical and industrial purposes. The thickness of lead necessary to shield a 400,000-volt X-ray tube adequately is of the order of $\frac{5}{8}$ inch. It is obvious that the weight and cost of such a room assumed prohibitive proportions. Massive walls of concrete were suggested as a substitute for lead. Although these had the advantage of being part of the building structure, concrete nevertheless intro-

**Increasing industrial uses of multimillion-volt X-rays has focused attention on protection against radiation hazards. In current Bureau tests, concrete slabs (4), each weighing 2½ tons and measuring 6 inches in thickness are placed one at a time over the mouth of the radiation pit (5). By varying concrete layers between target and pit, the effectiveness of concrete as a protective barrier may be determined. An ionization chamber can be moved by remote control to measure radiation at any point in the pit.**

duced serious engineering problems because of its weight.

It was not until the Bureau undertook a comprehensive study of concrete for protection that the economics of such protection could be reduced to a reasonable scale. Prior to this, concrete walls were simply installed by guess method. As a result of Bureau studies, it was possible to decrease the requisite wall thickness substantially and still maintain a high level of safety. It was further found that, by placing certain restrictions on the direction in which the X-ray beam might be pointed and yet not interfere with the treatment requirements, additional substantial economies in protection could be introduced. Studies were made to determine exactly how much protection would be required in a wall or floor that was not subjected to the primary X-ray beam, but was subjected only to the X-rays that were scattered from the material under irradiation.

By the time that X-ray voltages had gone up to 1 and 2 million volts, the protection problem again became critical. Studies were undertaken along the same line as for 400,000 volts, and it was determined that in most cases all secondary X-rays could be satisfactorily stopped by an ordinary reinforced concrete wall, varying in thickness from 8 to 12 inches. To cut off the direct beam would require from 12 to 18 inches of concrete, but it was usually possible to minimize this extra thickness of protective concrete by restricting the direction of the X-ray beam.

Early Bureau findings have been incorporated in the construction of the present high-voltage laboratory, which has X-ray tubes operating at 1½ million volts. These plans of course had to be made in advance, but after the building was completed careful radiation hazard surveys have shown that direct applications of the protection principles for concrete have provided a wide margin of safety.

With the advent of the betatron operating at from 50 million to 300 million volts, scientists are confronted with a new series of X-ray problems. Although protection from the direct beam of X-rays at 50 million volts is very important, there are other simplifying factors in the problem. For example, as science goes to higher and higher voltages in the production of X-rays, the X-rays themselves tend more and more to merge in a single direction from the target. Thus the X-ray beam emerging from a 50 million-volt betatron covers the very small angle of only 6 or 8 degrees. For this beam alone, very great thicknesses of concrete may be required. On the other hand, X-rays that are scattered sideways out of this beam are of much less penetrating character, and as a consequence it may be found that the protection problem to the side of the beam may not be seriously different from that at 1 or 2 million volts.

These problems, however, are all uncertain at the present time, and it is the responsibility of the Bureau to set up the necessary experimental arrangements to evaluate them properly. Again, a solution must be found that is safe and yet provides the maximum economy, as the cost of protection alone in a 50-million-volt medical or industrial installation may represent half to three-quarters of the total cost.

# Tables of Chemical Thermodynamic Properties

The first tables in a new and comprehensive compilation of "Tables of Selected Values of Chemical Thermodynamic Properties" have just been announced by the National Bureau of Standards. These tables, compiled by the Bureau in cooperation with the Office of Naval Research, United States Navy Department, are prepared in loose-leaf form to facilitate adding supplements as they become available. It is planned to have additional tables issued each quarter.

The selected values of thermodynamic properties are presented in three series of tables. These tables are internally consistent in that all the known physical and thermodynamic relations existing among the properties in the several tables are satisfied by the tabulated values of these properties. The three series are as follows:

*SERIES I*

Heat of Formation at 25° C
Free Energy of Formation at 25° C
Entropy at 25° C
Logarithm of the Equilibrium Constant of Formation at 25° C
Heat of Formation at 0° K
(Arranged by groups of compounds)

*SERIES II*

Heat, Temperature, and Entropy of Transition
Heat, Temperature, and Entropy of Fusion
Heat, Temperature, and Entropy of Vaporization
(Including values of pressure as appropriate)
(Arranged by groups of compounds)

*SERIES III*

Heat of Formation
Free Energy of Formation
Logarithm of the Equilibrium Constant of Formation
Free Energy Function
Heat Content (or Enthalpy) Function
Entropy
Heat Content or Enthalpy
Heat Capacity
(From 0° K to high temperatures)
(Values tabulated for individual compounds)

Complete references to the data from which the selected values have been obtained will be published later. References to experimental data that may have been overlooked in the selection of values of a given property for a given compound, as well as contributions of unpublished data and reprints of published work, will be greatly appreciated.

One set of these tables, with supplements as issued, is being furnished to each University department of physics, chemistry, or engineering. U. S. Government laboratories, research institutions, and industrial laboratories may obtain one set each on request to the National Bureau of Standards, Washington 25, D. C.

# Mercury-Balance Method for the Measurement of Gel Strength

An improved method for more precise measurements of the tensile strength of gels [3] has been developed by W. J. Hamer of the Bureau's electrochemistry laboratory. This procedure, which gives the strength or "yield value" and resistance to deformation of the material under study, involves essentially the determination of the shearing force necessary to fracture the gel as given by the weight of mercury required to pull a standard disk from the gel in which it is embedded.

In many applications of gelatin, glue, starch, agar, and various gums, it is important to know the tensile strength of the gel or jelly. This property was originally measured by comparison with a set of arbitrary standards on the basis of the resistance offered by the gel to finger pressure. Although this procedure was undoubtedly satisfactory for many commerical purposes, there was need for a more objective method. Such a method is very important, for example, in studies of gelatin coatings in photographic operations, particularly with the increasing use of flexible film supports instead of rigid glass bases.

The new Bureau development represents a precise objective method for gel-strength measurement. Although this procedure was designed specifically for the study of the paste walls of dry cells, it may be applied to all types of gels and used in the study of food starches, in following small changes in starch pastes for use in adhesives, and in many other applications.

The method involves the balancing of the cohesive forces of the gel against a gradually increasing quantity of mercury until the yield point is reached and the gel is broken. A brass disk is suspended in a beaker containing the gel by means of a wire attached to one beam of an analytical balance. An adjustable platform over the balance pan supports the beaker. The gel is covered with liquid petrolatum to prevent "skin" formation. Mercury is added from a burette at a constant rate to a container on the other pan. Different flow rates are made possible by use of a removable capillary tip held to the burette by a rubber tube. The capillary tip is calibrated before use by weighing the mercury it delivers in a given time. As the mercury is added, the height of the platform is changed manually, so that the pointer of the balance is always kept at zero. A vertical scale indicating the height of the adjustable platform is read at frequent regular intervals until the gel is broken.

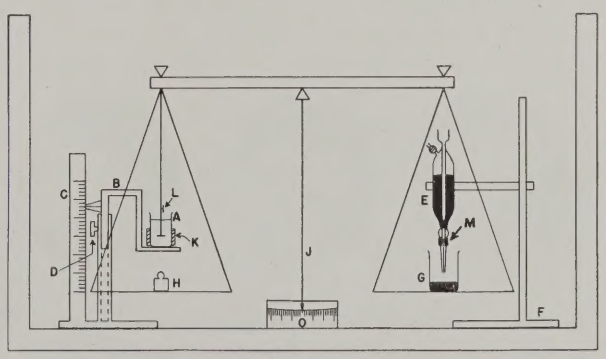
The weight of mercury required to produce a fracture in the gel, divided by the area of the brass disk gives gel strength in grams per square centimeter. The rate of change in height of the adjustable platform gives an indication of the resistance the gel offers to deformation. A fast rate means that the gel is easily deformed, whereas a slow rate indicates that the gel resists deformation and is probably quite rigid. Although rigid gels usually offer high resistance to deformation, this is not always true.

This method as a precision greater than 0.5 percent. Its limitations lie more in the reproducibility with which gels can be prepared than in errors inherent in the method. It may be applied to all types of gels, whether they are of the reversible (gelatin) or irreversible (starch) type.

Gel-strength measurements are used in the control of several industrial processes, such as the manufacture of adhesives, pectin jellies, starch sponges, and dextrins; in the preparation of foods; and to a more limited extent in the paper and textile industries. They are also frequently used in studies of the granular structure of starches and in determinations of the effects of acids, heat, aging, alcohols, polyhydrols, and other physical or chemical factors on the elasticity of starch and gelatin gels.

The paste wall of most dry cells consists of a mixture of starch and flour in the form of an aqueous gel. Recently the Bureau has made studies of the paste-wall separator of dry cells of the Leclanche type. Gel-strength measurements were used to differentiate walls of different starches, to find out whether the paste reacted with the other constituents of the cell, and to determine the effects of chemical and physical modifications on the strength of the paste wall. The results were correlated with the shelf life of dry cells made with the various types of paste walls.

The characteristics of the paste walls are often described in terms of gelling temperature of the paste or the variety and amount of starch and flour used in its preparation. Frequently the viscosity of the paste is also used for this purpose. For quantitative results, however, a more sensitive method is needed, preferably one that may be used with the gels themselves rather than the pastes from which they are formed. Numerous procedures have been proposed for objective measurements of gel strengths. Several of these are limited




**Gel strength is accurately determined through measurement of the weight of mercury, *G*, required to pull a standard disk from the gel in beaker, *A*. In addition, the rate of change in height of the adjustable platform, *B*, necessary to keep pointer, *J*, at zero gives an indication of resistance to deformation.**

in application, because they depend on the breaking or compression of the surface of the gel and are thus subject to errors owing to skin formations of variable thickness. Furthermore, many methods give a measure of different characteristics of a gel depending upon the point of view of the experimenter. For example, they may indicate rigidity, elasticity, plastic viscosity, or resistance to some force such as cutting action or torsion. The new method developed at the Bureau gives consistent results, well adapted to use in the Bureau's studies of the paste walls of dry cells.

[3] For further details of this method, see Research Paper RP1810, An improved method for measurement of gel strength and data on starch gels, W. J. Hamer, J. Research NBS **39,** 29 (1947).

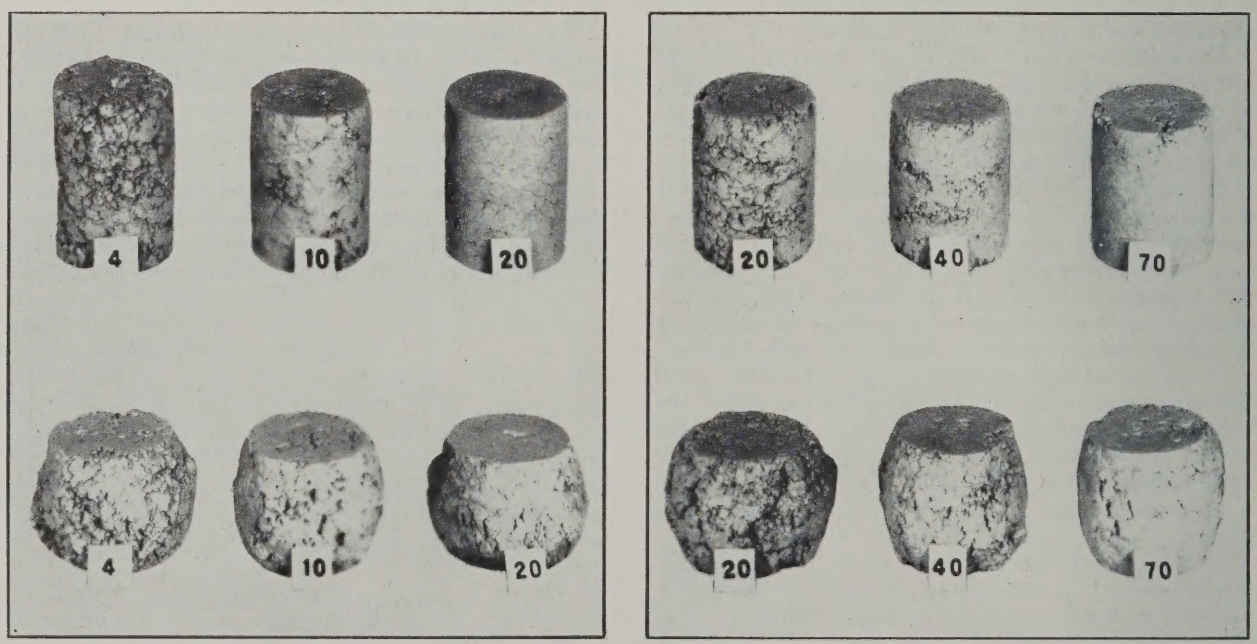
# Workability Index of Fireclay Refractories

During the past two decades, fireclay plastic refractories have gained wide use in industry for such applications as furnace linings, forming into refractory shapes, and quick repair jobs. An important requirement of this commodity, therefore, is that it flow without cracking while being rammed into molded shapes or furnace walls, and yet not deform under its own weight during subsequent drying.

Because of the numerous variables in determination of the workability of fireclay plastic refractories that affect their acceptability, the refractories laboratory of the Bureau has conducted an extensive investigation of the reliability of the test method now accepted as standard. Results of tests on nine plastic refractories by R. A. Heindl and W. L. Pendergast have provided basic data for use in revision of the method and technics of testing such plastics that will furnish a truer indication of workability, as a basis for acceptance or rejection.

A measure of the facility with which plastic refractary material can be rammed into place as determined with a specified sand rammer has been designated "workability index" by the American Society for Testing Materials. The instrument used for determining the index, the "sand rammer", is so called because of its use in the testing of foundry sands. The first step in the ASTM test of plastic refractories involves the molding of a standard 300-gram cylindrical test specimen with 20 impacts (10 on each end) of a 14-pound weight. Immediately after removing the specimen from the mold, and measuring its length, it is subjected to three additional impacts after which it is again measured. The plastic deformation computed from the difference in lengths and expressed in percent, is the workability index. The specified permissible range of this index is from 15 to 35 percent.

The first of the variables investigated by the Bureau




**Specimens of two of nine brands of fireclay plastic refractories subjected to "workability index" tests by the Bureau's refractories laboratory illustrate the spread of test results obtained by the present standard method. Labels refer to number of impacts for forming specimens. Maximum bulk density is not always reached with the 20 impacts specified by the ASTM method. One group of samples (upper left) reached this condition before 20 impacts (lower right) whereas the other group (upper right) did not reach maximum bulk density until 70 impacts (lower right). The latter material should be rejected although the ASTM workability index indicated acceptability.**

was the mounting for the sand rammer. The results obtained with four types of mounting indicate that the rammer should be solidly mounted on a nonvibrating stone or concrete block in order that a minimum amount of the work performed would be absorbed by the support. Lubrication of the mold not only improved the appearance of the test specimens, but decreased the workability index as much as 20 percent. This was due to the decrease in friction between the wall and the plastic refractory—especially when the moisture content was low.

Obtaining a cored sample with a steel tube, instead of a "grab sample", gave information on the extent of moisture variation in the plastic from top to bottom and across the 200-pound container. For example, one plastic had an over-all moisture variation from 8.1 to 9.9 percent, with an average of 8.4 percent. The method of obtaining the sample as well as that of filling the mold resulted in pronounced differences in the magnitude of the workability index. The interior of the steel mold for forming the test cylinder should be machined smooth and a ¼-inch steel disk, made to fit the interior snugly, placed at each end of the test specimen during forming. The use of the disks prevents feather-edging of the specimen and possible loss of material.

The 20 impacts specified in the ASTM method of test for molding a plastic refractory do not always result in the specimen reaching maximum bulk density. Some of the plastic refractories investigated by the Bureau reached their maximum bulk density (or minimum height) before 20 impacts, whereas others did not reach the same condition until 70 impacts. The further the specimen is from maximum density the greater the possibility that the workability index may be affected by disruptive failures rather than by plastic deformation. The index, therefore, may be misleading as it would indicate a workability higher than warranted. For instance, using the standard test method, an index of 24 was obtained for one of the specimens, signifying acceptable material. However, after 70 impacts the index was 13, indicating that the material should be rejected, which should have been the case initially as it was too stiff for satisfactory use.

As a result of the Bureau investigations, it has been recommended that the workability of a plastic refractory be based on the amount of work required to reach maximum bulk density rather than on a deformation test. In addition, the findings also indicate the need for the standardization of the method of test.

# Gas Economizers Prove of Little Value

Engineers at the National Bureau of Standards have recently tested a number of gas economizers, devices alleged to reduce the fuel consumption of the average automobile engine. The postwar "economizers" tested do not differ radically from prewar models, although their names tend to keep up with the latest in engineering and are often designated as electronic or supercharger devices.

Bureau engineers, who have been testing these devices for a number of years, report that they have never tested an economizer that had any basic value. Some may actually effect a small amount of fuel saving by adding additional air to the gas-air mixture prepared in the carburetor, but this job is done more effectively by carburetor adjustment. Most others tested showed no measurable effect whatsoever on either gas consumption or engine performance.

Of the devices that actually cause a change in engine operation, the basic types are those that lean the air-fuel ratio by letting more air into the carburetor or by adding air as the fuel mixture passes from the carburetor to the intake manifold. A device recently tested was made to take the place of the air cleaner—an important long-life feature of modern automobiles. It was essentially a funnel that faced the front of the car so that when the car was in motion extra air would flow into the carburetor. Test results showed that the amount of fuel saved using this funnel with a normal carburetor adjustment was small and engine performance was below normal. Although the device might result in a saving of fuel in a car with too rich a mixture, the saving is obtained more easily by a simple carburetor adjustment.

Engine tests have shown that the mixture that gives maximum engine power contains about 12 pounds of air to 1 pound of fuel, while the most economical mixture is around 17 pounds of air to 1 of fuel. The modern carburetor, a precision piece of equipment that represents extensive research on the part of automobile manufacturers, is designed to feed a rich mixture to the cylinders when the engine is called upon to develop high power and a leaner mixture for normal driving in high gear.

Because the majority of drivers are interested in top performance rather than maximum economy, carburetors are usually set a little on the rich side. However, most dealers and garages carry a lean carburetor jet which, when installed, may save up to 5 percent on gas for the driver who does not object to a lowered rate of pick-up and engine sluggishness. A competent mechanic should be consulted on the choice of the proper jet because it is possible to lean the gas-air mixture to the point where cylinder firing becomes so inefficient that an actual increase in fuel consumption results.

Of the devices that produce no measurable effect, a recent example was an "electronic gas saver" which, by utilizing an electric spark, was said to produce ozone that would boost power. The amount of ozone produced was so small that it could not be measured which was a negative virtue as ozone in quantity has been found to be a powerful knock inducer. The claim for another type of economizer, which fitted on the end of the exhaust pipe, was that, by reducing back pressure in the exhaust line, it would cause a more complete elimination of the burned fuel in the cylinder and in-

crease the efficiency of succeeding charges from the carburetor. In testing this device in the laboratory, a pressure drop was built up at the end of the exhaust line that was more than 30 times that which would be met anywhere outside the laboratory before a noticeable change in gas use occurred.

# NBS Publications

## *Periodicals* [4]

Journal of Research of the National Bureau of Standards, volume **39**, number 1, July 1947. (RP1807 to RP1815, inclusive).
Technical News Bulletin, volume **31**, number 7, July 1947. 10 cents.
CRPL–D35. Basic Radio Propagation Predictions for October 1947. Three months in advance. Issued July 1947. 15 cents.

## *Nonperiodical*

### Research Papers [4,5]

RP1796. An improved ice calorimeter—the determination of its calibration factor and the density of ice at 0° C. Defoe C. Ginnings and Robert J. Corruccini. Price 10 cents.
RP1797. Enthalpy, specific heat, and entropy of aluminim oxide from 0° to 900° C. Defoe C. Ginnings and Robert J. Corruccini. Price 10 cents.
RP1798. Transmission measurements with the Beckman quartz spectrophotometer. Kasson S. Gibson and Margaret M. Bal-Com. Price 10 cents.
RP1799. Mass spectrometer study of rare gases. Vernon H. Dibeler, Fred L. Mohler, and Robert M. Reese. Price 15 cents.
RP1800. Hydrocarbons in the 102° to 108° C fraction of petroleum. Augustus R. Glasgow, Jr., Charles B. Willingham, and Frederick D. Rossini. Price 10 cents.
RP1801. Heats of combustion and solution of liquid styrene and solid polystyrene, and the heat of polymerization of styrene. Donald E. Roberts, William W. Walton, and Ralph S. Jessup. Price 10 cents.
RP1802. Torsion of a rubber cylinder. Ronald S. Rivlin. Price cents.
RP1803. Compensation of the aperture ratio markings of a photographic lens for absorption, reflection, and vignetting losses. Irvine C. Gardner. Price 10 cents.
RP1804. Heat capacities of gaseous oxygen, isobutane, and 1-butene from −30° to +90° C. Paul F. Wacker, Ruth K. Cheney, and Russell B. Scott. Price 10 cents.
RP1805. Heats of solution of solid solutions of hexacalcium dialumino ferrite and dicalcium ferrite. Edwin S. Newman. Price 5 cents.
RP1806. Relative thickness of lead, concrete, and steel required for protection against narrow beams of X-rays. George Singer, Harold O. Wykoff, and Frank H. Day. Price 10 cents.

### Simplified Practice Recommendations [4]

R179–46. Structural insulating board. (Supersedes R179–42.) 5 cents.
R197–46. Glass containers for maraschino cherries. (Supersedes R197–42.) 5 cents.
R31–47. Loaded paper shot shells. (Supersedes R31–45.) 5 cents.
R62–47. Metallic cartridges. (Supersedes R62–45.) 5 cents.
R97–47. Bell-bottom screw jacks. (Supersedes R97–42.) 5 cents.

### Commercial Standards [4]

CS139–47. Work gloves. 10 cents.

### Circulars [4]

C459. Announcement of changes in electrical and photometric units. 5 cents.

### Miscellaneous [4]

M186. Report of the thirty-second National Conference on Weights and Measures. 40 cents.

### Letter Circulars [6]

LC860. Standards and specifications for building and construction materials, fixtures, supplies, and equipment. (Supersedes LC808.)
LC861. Inside wall paint for chemical laboratories. (Fume-resisting enamel paint.) (Supersedes LC489.)
LC862. Fire resistance and fire prevention. (Supersedes LC661.)
LC863. Condensation in buildings.
LC864. Effect of changes in electrical and photometric units on previously assigned values for standard lamps.
LC865. Dampness in masonry walls above grade. (Supersedes LC721.)
LC866. House plan services. (Supersedes LC829.)

## *Articles by Bureau Staff Members in Outside Publications* [7]

Rapid determination of small amounts of carbon dioxide. Martin Shepherd. Analytical Chemistry (332 West 42d St., New York 18, N. Y.) **19,** No. 2, 77 (February 1947).
Mangabeira latex and rubber. Norman Bekkedahl and Waldemar Saffioti. Rubber Age (250 West 57th St., New York 19, N. Y.) **60,** No. 5, 553 (February 1947).
Laboratory testing of rubber for cut-growth. W. L. Holt and E. O. Knox. Rubber Age (250 West 57th St., New York 19, N. Y.) **60,** No. 6, 689 (March 1947).
Rubber research and technology at the National Bureau of Standards. L. A. Wood. India Rubber World (386 4th Ave., New York 16, N. Y.) **115,** No. 6, 789 (March 1947).
Natural rubbers: A general summary of their composition, properties, and uses. Norman Bekkedahl. India Rubber World (386 4th Ave., New York 16, N. Y.) **116,** No. 1, 57 (April 1947).
Torsion of a rubber cylinder. R. S. Rivlin. Journal of Applied Physics (American Institute of Physics, 57 East 55th St., New York 22, N. Y.) **18,** No. 5, 444 (May 1947).
How simplified practice aids the construction industry. E. W. Ely. Domestic Commerce (U. S. Department of Commerce, Washington 25, D. C.) **35,** No. 5, 19 (May 1947).
Simplified practices assist in distribution. E. W. Ely. Domestic Commerce (U. S. Department of Commerce, Washington 25, D. C.) **35,** No. 6, 11 (June 1947).
Electron configurations of "rare earth" elements. W. F. Meggers. Science (1313 Massachusetts Ave NW., Washington 5, D. C.) **105,** No. 2733, 514 (May 1947).

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[4] Send orders for publications under this heading only to the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Annual subscription rates: Journal of Research, $4.50 (foreign, $5.50); Technical News Bulletin, $1.00 (foreign, $1.35); Basic Radio Propagation Predictions, $1.50 (foreign, $2.00). Single copy prices of publications are indicated in the lists.
[5] Reprints from June Journal of Research.
[6] Available on request from the National Bureau of Standards, Washington 25, D. C. Letter Circulars are prepared to answer specific inquiries addressed to the Bureau, and are sent only on request to persons having a definite need for the information. The Bureau cannot undertake to supply lists or complete sets of Letter Circulars or send copies automatically as issued.
[7] These publications are not available from the Government. Requests should be sent direct to the publishers.



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